Process for producing a catalyst for gas-phase oxidations

Description

5 Process for producing a catalyst for gas-phase oxidations, and also the use of the catalyst for the catalytic gas-phase oxidation of aromatic hydrocarbons to carboxylic acids and/or carboxylic anhydrides, in particular for the preparation of phthalic anhydride from o-xylene, naphthalene or mixtures thereof.

10 Many carboxylic acids and/or carboxylic anhydrides are prepared industrially by the catalytic gas-phase oxidation of aromatic hydrocarbons such as benzene, the xylenes, naphthalene, toluene or durene in fixed-bed reactors. In this way, it is possible to obtain, for example, benzoic acid, maleic anhydride, phthalic anhydride, isophthalic acid, terephthalic acid or pyromellitic anhydride. In general, a mixture of an oxygen-
15 containing gas and the starting material to be oxidized is passed through tubes in which a bed of a catalyst is located. To regulate the temperature, the tubes are surrounded by a heat transfer medium, for example a salt melt.

 Catalysts which have been found to be useful for these oxidation reactions are coated
20 catalysts in which the catalytically active composition is applied in the form of a shell to an inert support material such as steatite. Compositions of differing catalytic activity can be applied in one or more layers. The catalytically active constituent of the catalytically active composition of these coated catalysts is generally titanium dioxide together with vanadium pentoxide. Furthermore, small amounts of many other oxidic
25 compounds which act as promoters to influence the activity and selectivity of the catalyst can be present in small amounts in the catalytically active composition.

 To produce such coated catalysts, an aqueous suspension of the constituents of the active composition and/or their precursor compounds or sources is sprayed onto the
30 support material at elevated temperature until the desired proportion by weight of active composition in the total catalyst has been achieved. Fluidized-bed apparatuses are particularly suitable for this purpose. In these apparatuses, the support material is fluidized in an ascending stream of gas, in particular air. The apparatuses usually comprise a conical or spherical container into which the fluidizing gas is introduced
35 from below or from above via a central tube. The suspension is sprayed into the fluidized bed from above, from the side or from below by means of nozzles. The use of a guide tube arranged centrically or concentrically around the central tube is advantageous. Within the guide tube, the gas velocity is higher and transports the support particles upward. In the outer ring, the velocity is only slightly above the
40 loosening velocity. The particles are therefore transported vertically in a circular motion.

 A suitable fluidized-bed apparatus is described, for example, in DE-A 40 06 935.

To improve the quality of the coating, it has become established practice in industry to add organic binders, preferably copolymers, advantageously in the form of an aqueous suspension, of vinyl acetate-vinyl laurate, vinyl acetate-acrylate, styrene-acrylate or vinyl acetate-ethylene, to the suspension. Furthermore, the addition of binder has the
5 advantage that the active composition adheres well to the support, so that transport and installation of the catalyst are made easier.

In the thermal treatment at temperatures of from > 80 to 450°C, the binder is driven off from the applied layer by thermal decomposition and/or combustion. The thermal
10 treatment is usually carried out in situ in the oxidation reactor.

The quality of the supported catalysts obtainable in this way, in particular the coating quality, depends decisively on operating parameters of the fluidized-bed apparatus, in particular on the total mass of support material in the apparatus, the binder content of
15 the suspension sprayed in, the flow rate and the temperature of the gas stream blown in for fluidization and the rate at which the suspension is sprayed onto the fluidized inert support. The setting of the most important operating parameters of the fluidized-bed apparatus for coating the support materials is in the prior art carried out by means of costly empirical trials which have to be carried out on the production scale since
20 scale-up from the laboratory or pilot plant scale to the production scale is virtually impossible because of the lack of satisfactory theoretical models.

WO 98 14274 describes a process for producing a supported catalyst in a fluidized-bed apparatus, in which a less than 100 μm layer of an active composition in aqueous
25 suspension is applied to an inert support having a diameter of 5 μm – 20 mm.

WO 02 096557 describes a process for producing supported metallic nanoparticles as catalysts in a fluidized-bed apparatus.

30 US 4 977 126 describes a process for preparing supported catalysts in a fluidized-bed apparatus, in which the catalysts comprise a layer of metallic cobalt on oxide supports.

FR 2 791 905 describes a process for producing supported catalysts, in which the suspension comprises fine particles having a diameter of 10-100 μm and a density of
35 more than 1000 kg/m^3 and contains about 30% of coarser particles having diameters of 0.4 - 1 mm.

However, these documents describe neither catalysts for gas-phase oxidations nor the coating of rings.
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It is therefore an object of the invention to provide a process for producing a catalyst for gas-phase oxidations in a fluidized-bed apparatus, in which a uniform and reproducible coating of a support material is obtained without costly preliminary experiments.

It has surprisingly been found that this object can be achieved when the amount of support material weighed into the apparatus, the throughput and the temperature of the gas stream introduced, and also the rate of introduction and the binder content of the suspension sprayed in are selected within particular prescribed ranges in such a way

5   that these parameters obey a simple empirically determined mathematical relationship.

The invention relates to a process for producing a catalyst for gas-phase oxidations, which comprises weighing a particulate inert support having a total mass of $M_{support}$ into a fluidized-bed apparatus, providing at least an aqueous suspension of a catalytically

10   active material or sources therefor and a binder having a binder content of B_{susp}, fluidizing the inert support by introduction of a gas stream heated to a temperature of T_{gas} at a flow rate of Q_{gas}, and spraying the suspension at a rate of Q_{susp} onto the fluidized inert support.

15   According to the invention, Q_{gas}, Q_{susp}, B_{susp}, $M_{support}$ and T_{gas} are selected within the ranges

$$3000 \leq Q_{gas} \text{ [m}^3\text{/h]} \leq 9000, \qquad 1000 \leq Q_{susp} \text{ [g/min]} \leq 3500,$$
$$2 \leq B_{susp} \text{ [\% by weight]} \leq 18, \qquad 60 \leq M_{support} \text{ [kg]} \leq 240.$$
$$75 \leq T_{gas} \text{ [°C]} \leq 120$$

20   so that a parameter K defined as

$$K = 0.020\, Q_{gas} - 0.055\, Q_{susp} + 7.500\, B_{susp} - 0.667\, M_{support} + 2.069\, T_{gas} - 7$$

satisfies the relationship $127.5 \leq K \leq 202$.

When the operating parameters satisfy this relationship, high-quality layers are

25   produced. In particular, the formation of twins, i.e. support bodies which adhere to one another, for example as a result of insufficient drying or too much binder, is avoided. In addition, very little if any attrition due to flaking-off of layers occurs. The layers themselves are also more uniform, both when the supports are coated with one layer and with two layers, than in a coating process in which one or more parameters do not

30   obey the above relationship.

The mechanical stability of the layer on the support is also improved.

In the process of the invention, the application of the layer(s) of the coated catalyst is

35   carried out, for example, by spraying a suspension of TiO_2 and V_2O_5, which comprises, if appropriate, sources of the promoter elements specified below, onto the fluidized support. The catalytically active composition in the calcined state preferably comprises, based on the total amount of catalytically active composition, from 1 to 40% by weight of vanadium oxide, calculated as V_2O_5, and from 60 to 99% by weight of titanium

40   dioxide, calculated as TiO_2.

As vanadium source, preference is given to using pulverulent vanadium pentoxide (V^{5+}) or dissolved vanadium, e.g. vanadyl oxalate (V^{4+}). Suitable starting compounds for the element vanadium are, for example, vanadium oxides such as vanadium pentoxide

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(V_2O_5), vanadates such as ammonium metavanadate, vanadium oxysulfate hydrate, vanadyl acetylacetonate, vanadium halides such as vanadium tetrachloride (VCl_4) and vanadium oxyhalides such as $VOCl_3$. In addition, it is also possible to make use of vanadium starting compounds in which the vanadium is present in the oxidation state

5 +4 or which comprise vanadium in the oxidation state +5 and various reducing agents (e.g. NH_4^+ or its decomposition product NH_3) which can reduce V^{5+} to V^{4+}. Such a reducing agent can also be oxalic acid, oxalate, hydrazine dihydrochloride, hydrazine sulfate, hydrazine (monohydrate), hydroxylamine, hydroxylamine hydrochloride or salts thereof.

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The catalytically active composition can further comprise up to 1% by weight of a cesium compound, calculated as Cs, up to 1% by weight of a phosphorus compound, calculated as P, and up to 10% by weight of antimony oxide, calculated as Sb_2O_3.

15 Apart from the optional additives cesium and phosphorus, the catalytically active composition can in principle contain small amounts of many other oxidic compounds which act as promoters to influence the activity and selectivity of the catalyst, for example reducing or increasing its activity. Examples of such promoters are the alkali metal oxides, in particular the abovementioned cesium oxide and also lithium,

20 potassium and rubidium oxide, thallium(I) oxide, aluminum oxide, zirconium oxide, iron oxide, nickel oxide, cobalt oxide, manganese oxide, tin oxide, silver oxide, copper oxide, chromium oxide, molybdenum oxide, tungsten oxide, iridium oxide, tantalum oxide, niobium oxide, arsenic oxide, cerium oxide. In general, cesium is used as promoter from this group.

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Among the promoters mentioned, preferred additives also include the oxides of niobium and tungsten in amounts of from 0.01 to 0.50% by weight, based on the catalytically active composition. As additives which increase the activity but decrease the selectivity, particular mention may be made of oxidic phosphorus compounds, in

30 particular phosphorus pentoxide.

Before carrying out the coating procedure, the suspension is preferably stirred for a sufficiently long time, e.g. from 2 to 30 hours, in particular from 12 to 25 hours, to break up agglomerates of the suspended solids and to produce a homogeneous suspension.

35 The suspension typically has a solids content of from 20 to 50% by weight. The suspension medium is generally aqueous, e.g. water itself or an aqueous mixture with a water-miscible organic solvent such as methanol, ethanol, isopropanol, formamide and the like. If the first or second suspension comprises TiO_2 and V_2O_5 particles as catalyst particles, preference is given to at least 90% by volume of the V_2O_5 particles

40 having a diameter of 20 µm or less and at least 95% by volume of the V_2O_5 particles having a diameter of 30 µm or less.

In general, organic binders, preferably copolymers, advantageously in the form of an aqueous dispersion, of vinyl acetate-vinyl laurate, vinyl acetate-acrylate, styrene-

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acrylate and vinyl acetate-ethylene, are added to the suspension. The binders are commercially available as aqueous dispersions having a solids content of, for example, from 35 to 65% by weight. The amount of such binder dispersions used is, according to the invention, from 2 to 18% by weight, based on the weight of the suspension.

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When the catalysts support is coated with the catalytically active composition, coating temperatures of from 75 to 120°C are employed according to the invention, with coating being able to be carried out under atmospheric pressure or under reduced pressure.

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The layer thickness of the catalytically active composition is generally from 0.02 to 0.25 mm, preferably from 0.05 to 0.20 mm. The proportion of active composition in the catalyst is usually from 5 to 25% by weight, mostly from 7 to 15% by weight.

15   Thermal treatment of the resulting precatalyst at temperatures of > 80 to 450°C drives the binder off from the applied layer as a result of thermal decomposition and/or combustion. The thermal treatment is preferably carried out in situ in the gas-phase oxidation reactor.

20   The parameter K is preferably in a range $136.0 \leq K \leq 193.5$ and

$4500 \leq Q_{gas} \,[m^3/h] \leq 7500,$ $1500 \leq Q_{susp} \,[g/min] \leq 3000,$
$5 \leq B_{susp} \,[\% \text{ by weight}] \leq 15,$ $100 \leq M_{support} \,[kg] \leq 200,$ and
$80 \leq T_{gas} \,[°C] \leq 115.$

25   The parameter K is particularly preferably in a range $143 \leq K \leq 184.5$ and

$5500 \leq Q_{gas} \,[m^3/h] \leq 6500,$ $2000 \leq Q_{susp} \,[g/min] \leq 2500,$
$6 \leq B_{susp} \,[\% \text{ by weight}] \leq 11$ $120 \leq M_{support} \,[kg] \leq 180,$
$90 \leq T_{gas} \,[°C] \leq 115.$

30   To fluidize and heat the bed of support material in the fluidized-bed apparatus, it is possible to use any gas or gas mixture which is inert under the operating conditions. However, the gas introduced is advantageously air, which makes particularly inexpensive operation of the plant possible.

35   The catalytically active composition can also be applied in two or more layers. The layers preferably have different selectivities and activities. For example, the inner layer or inner layers can have an antimony oxide content of up to 15% by weight and the outer layer can have an antimony oxide content which is from 50 to 100% lower. For example, the inner and outer layers may have different phosphorus contents. To

40   prepare catalysts having two layers according to the invention, a second aqueous suspension is prepared from catalytically active material and is sprayed onto the fluidized support which has been coated with the first suspension.

As inert support material, it is possible to use virtually all support materials of the prior

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art, as are used advantageously in the production of coated catalysts for the oxidation of aromatic hydrocarbons to aldehydes, carboxylic acids and/or carboxylic anhydrides. Support materials used are, for example, silica (SiO_2), porcelain, magnesium oxide, tin dioxide, silicon carbide, rutile, alumina (Al_2O_3), aluminum silicate, steatite (magnesium

5   silicate), zirconium silicate, cerium silicate or mixtures of these support materials. The support material is generally nonporous. For the purposes of the present invention, the expression "nonporous" means "nonporous except for technically insignificant amounts of pores", since a small number of pores may be unavoidably present under industrial conditions in a support material which should ideally contain no pores. Advantageous

10   support materials are, in particular, steatite and silicon carbide. The shape of the support material is generally not critical for the precatalysts and coated catalysts according to the invention. For example, catalyst supports in the form of spheres, rings, pellets, spirals, tubes, extrudates or crushed material can be used. The dimensions of these catalyst supports correspond to those of catalyst supports customarily used for

15   producing coated catalysts for the gas-phase partial oxidation of aromatic hydrocarbons. Preference is given to using steatite in the form of spheres having an external diameter of from 0.5 to 10 mm or rings having an external diameter of from 3 to 15 mm.

20   The process of the invention is particularly preferably carried out in a fluidized-bed apparatus which is a container for accommodating the particulate support in whose lower region a dish-like depression is provided and which comprises a central tube for introducing the gas which extends essentially axially downward in the container and opens into the depression, an essentially annular deflection shield which is fixed to the

25   central tube in the upper region of the container and a guide ring which is located in the lower region of the container and surrounds the central tube essentially concentrically over part of its length and means for spraying-in the first and, if applicable, second suspension. Such a fluidized-bed apparatus is described, for example, in the German patent application DE 40 06 935. Commerically available fluidized-bed apparatuses

30   which are suitable for carrying out the process of the invention are, for example, the Kugel-Coater HKC 150 and HKC 200 from Hüttlin, Steinen, Germany.

The catalysts produced according to the invention are suitable in general for the gas-phase oxidation of aromatics C_6-C_{10}-hydrocarbons such as benzene, the xylenes,

35   toluene, naphthalene or durene (1,2,4,5-tetramethylbenzene) to carboxylic acids and/or carboxylic anhydrides such as maleic anhydride, phthalic anhydride, benzoic acid and/or pyromellitic anhydride. The invention therefore also provides for the use of the catalyst produced by the process of the invention for preparing phthalic anhydride from o-xylene, naphthalene or mixtures thereof.

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For this purpose, the catalysts produced according to the invention are placed in reaction tubes which are thermostated from the outside to the reaction temperature, for example by means of salt melts , and the salt bath temperatures of generally from 300 to 450°C, preferably from 320 to 420°C and particularly preferably from 340 to 400°C

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and at a gauge pressure of generally from 0.1 to 2.5 bar, preferably from 0.3 to 1.5 bar, are passed at a space velocity of generally from 750 to 5000 h^{-1}. The reaction gas supplied to the catalyst is generally produced by mixing a gas which comprises molecular oxygen and may, in addition to oxygen, further comprise suitable reaction

5   moderators and/or diluents such as steam, carbon dioxide and/or nitrogen with the aromatic hydrocarbon to be oxidized, with the gas comprising molecular oxygen generally being able to comprise from 1 to 100 mol%, preferably from 2 to 50 mol% and particularly preferably from 10 to 30 mol%, of oxygen, from 0 to 30 mol%, preferably from 0 to 10 mol%, of water vapor and from 0 to 50 mol%, preferably from 0

10 to 1 mol%, of carbon dioxide, balance nitrogen. To produce the reaction gas, the gas comprising molecular oxygen is generally loaded with from 30 g to 150 g of the aromatic hydrocarbon to be oxidized per standard m^3 of gas. It has been found to be particularly advantageous to use catalysts which differ in terms of the catalytic activity and/or the chemical make-up of their active composition in the catalyst bed. When

15 using two reaction zones, the catalyst employed in the first reaction zone, i.e. the reaction zone nearest the gas inlet for the reaction gas, usually has a somewhat lower catalytic activity than the catalyst present in the second reaction zone, i.e. the reaction zone nearest the gas outlet. In general, the reaction is controlled by means of the temperature setting so that the major part of the aromatic hydrocarbon present in the

20 reaction gas is reacted at maximum yield in the first zone. Preference is given to using three- to five-zone catalyst systems, in particular three- and four-zone catalyst systems.

The invention is illustrated by the following examples.

25 <u>Example 1</u> (single-layer catalyst on conventional support rings):

47.44 kg of anatase (BET surface area: 9 m^2/g), 20.34 kg of anatase (BET surface area: 20 m^2/g), 5.32 kg of vanadium pentoxide, 1.33 kg of antimony oxide, 0.30 kg of cesium carbonate were suspended in 195 l of deionized water and stirred for 18 hours

30 to obtain a homogeneous dispersion. 30.6 kg of an organic binder consisting of a copolymer of vinyl acetate and vinyl laurate in the form of a 50% strength aqueous dispersion were added to this suspension.

In a fluidized-bed apparatus (Hüttlin HKC 150), 60 kg of this suspension were sprayed
35 onto 150 kg of steatite (magnesium silicate) in the form of rings having dimensions of 7 mm × 7 mm × 4 mm (external diameter × height × internal diameter) and dried. The operating parameters were

Air flow:	6000 m^3/h
Introduction rate:	2250 g/min
Binder concentration:	10% by weight of the total suspension used
Weight of supports:	150 kg of steatite rings (7 mm × 7 mm × 4 mm)
Temperature of air fed in:	109°C

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The catalytically active composition applied in this way, i.e. the catalyst coating, comprised 7.12% by weight of vanadium (calculated as V_2O_5), 1.8% by weight of antimony (calculated as Sb_2O_3), 0.33% by weight of cesium (calculated as Cs) and 90.75% by weight of titanium dioxide after calcination at 450°C for one hour. The weight of the applied coating was 8.0% of the total weight of the finished catalyst.

The parameter K calculated from the equation in claim 1 is 188.5.

The amount of abraded material after a triple drop test was 25% by weight (after calcination at 450°C for one hour). In the drop test, about 50 g of catalyst (calcined after thermal treatment for one hour at 450°C) were allowed to drop through a 3 m long tube having an internal diameter of 25 mm. The catalyst falls into a dish standing under the tube, is separated from the dust formed on impact and is again allowed to drop through the tube. The total mass loss in the triple drop test based on the amount of active composition applied, which corresponds to 100%, is a measure of the abrasion resistance of the catalyst.

Comparative example 2:

The catalyst was prepared as in example 1, with the operating conditions of the fluidized-bed apparatus being set as follows:

Air flow:	6000 m³/h
Introduction rate:	2250 g/min
Binder concentration:	10% by weight of the total suspension used
Weight of supports:	150 kg of steatite rings (7 mm x 7 mm x 4 mm)
Temperature of air fed in:	70°C

The parameter K calculated from the equation in claim 1 is 107.8.

When the temperature of the air fed in was below the range according to the invention, many twin rings which obviously resulted from insufficient drying were found. The amount of abraded material after the triple drop test (drop test as in example 1) was 40%.

Comparative example 3:

The catalyst was prepared as in example 1, with the operating conditions of the fluidized-bed apparatus being set as follows:

Air flow:	6000 m³/h
Introduction rate:	2250 g/min
Binder concentration:	20% by weight of the total suspension used
Weight of supports:	150 kg of steatite rings (7 mm x 7 mm x 4 mm)

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Temperature of air fed in: 109°C

The parameter K calculated from the equation in claim 1 is 263.5.

5   At a binder concentration above the range specified according to the invention, many twin rings were also found. The amount of abraded material after the triple drop test (drop test as in example 1) was 40%.

Example 4 (single-layer catalyst on larger support rings):

10   150 kg of steatite in the form of rings having dimensions of 8 mm × 6 mm × 5 mm (external diameter × height × internal diameter) were heated in a fluidized-bed apparatus (Hüttlin HKC 150) and sprayed with 57 kg of a suspension comprising 140.02 kg of anatase having a BET surface area of 21 m^2/g, 11.776 kg of vanadium

15   pentoxide, 31.505 kg of oxalic acid, 5.153 kg of antimony trioxide, 0.868 kg of ammonium hydrogenphosphate, 0.238 g of cesium sulfate, 215.637 kg of water and 44.808 kg of formamide together with 33.75 kg of an organic binder consisting of a copolymer of acrylic acid-maleic acid (weight ratio = 75:25) until the weight of the layer applied was 10.5% of the total weight of the finished catalyst (after heat treatments at

20   450°C for one hour). The catalytically active composition applied in this way, i.e. the catalyst coating, comprised on average 0.15% by weight of phosphorus (calculated as P), 7.5% by weight of vanadium (calculated as V_2O_5), 3.2% by weight of antimony (calculated as Sb_2O_3), 0.1% of cesium (calculated as Cs) and 89.05% by weight of titanium dioxide. The operating conditions of the fluidized-bed apparatus were:

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Air flow: 6500 m^3/h
Introduction rate: 2250 g/min
Binder concentration: 7.5% by weight of the total suspension used
Weight of supports: 150 kg of steatite rings (8 mm × 6 mm × 5 mm)
30   Temperature of air fed in: 97°C

The parameter K calculated from the equation in claim 1 is 154.9. The amount of abraded material after the triple drop test (drop test as in example 1) was 5% by weight (after calcination at 450°C for one hour).

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Comparative example 5:

The catalyst was prepared as in example 4, with 19 kg of the suspension being sprayed and the operating conditions of the fluidized-bed apparatus being set as
40   follows:

Air flow: 650 m^3/h
Introduction rate: 2250 g/min
Binder concentration: 7.5% by weight of the total suspension used

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Weight of supports:	50 kg of steatite rings (8 mm × 6 mm × 5 mm)
Temperature of air fed in:	97°C

The parameter K calculated from the equation in claim 1 is 221.6.

In this comparative example in which the weight of support material was below the range specified according to the invention, many twin rings were once again found. The amount of abraded material after the triple drop test (drop test as in example 1) was 34%.

Comparative example 6:

The catalyst was prepared as in example 4, with the operating conditions of the fluidized-bed apparatus being set as follows:

Air flow:	6500 m³/h
Introduction rate:	900 g/min
Binder concentration:	7.5% by weight of the total suspension used
Weight of supports:	150 kg of steatite rings (8 mm × 6 mm × 5 mm)
Temperature of air fed in:	97°C

The parameter K calculated from the equation in claim 1 is 229.9.

At a rate of introduction of the suspension which was too low, many flaked coated catalysts were found. The amount of abraded material after the triple drop test (drop test as in example 1) was 51%.

Example 7: Two-layer catalyst

Suspension 1:
150 kg of steatite in the form of rings having dimensions of 8 mm × 6 mm × 5 mm (external diameter × height × internal diameter) were heated in a fluidized-bed apparatus (Hüttlin HKC 150) and sprayed with 24 kg of a suspension comprising 155.948 kg of anatase having a BET surface area of 21 m²/g, 13.193 kg of vanadium pentoxide, 35.088 kg of oxalic acid, 5.715 kg of antimony trioxide, 0.933 kg of ammonium hydrogenphosphate, 0.991 g of cesium sulfate, 240.160 kg of water and 49.903 kg of formamide together with 37.5 kg of an organic binder consisting of a copolymer of acrylic acid-maleic acid (weight ratio = 75:25).

Suspension 2:
150 kg of the coated catalyst obtained were heated in a fluidized-bed apparatus and sprayed with 24 kg of a suspension comprising 168.35 kg of anatase having a BET surface area of 21 m²/g, 7.043 kg of vanadium pentoxide, 19.080 kg of oxalic acid, 0.990 g of cesium sulfate, 238.920 kg of water and 66.386 kg of formamide together

with 37.5 kg of an organic binder consisting of a copolymer of acrylic acid-maleic acid (weight ratio = 75:25).

Operating conditions of the fluidized-bed apparatus during spraying-on of both layers:

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Air flow:	6500 m³/h
Introduction rates:	in each case 2250 g/min
Binder concentration:	7.5% by weight of the total suspension used
Weight of supports:	150 kg of steatite rings (8 mm x 6 mm x 5 mm)
Temperature of air fed in:	97°C

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The parameter K calculated from the equation in claim 1 is 154.9.

The weight of the layers applied was 9.3% of the total weight of the finished catalyst

15   (after heat treatment at 450°C for one hour). The catalytically active composition applied in this way, i.e. the catalyst coatings, was on average 0.08% by weight of phosphorus (calculated as P), 5.75% by weight of vanadium (calculated as V_2O_5), 1.6% by weight of antimony (calculated as Sb_2O_3), 0.4% by weight of cesium (calculated as Cs) and 92.17% by weight of titanium dioxide.

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The amount of abraded material after the triple drop test (drop test as in example 1) was 10% by weight (after calcination at 450°C for one hour).

Comparative example 8:

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A two-layer catalyst was prepared as in example 7, with the operating conditions of the fluidized-bed apparatus being set as follows:

Air flow:	2900 m³/h
Introduction rate:	2250 g/min
Binder concentration:	7.5% by weight of the total suspension used
Weight:	150 kg of steatite rings (8 x 6 x 5 mm)
Temperature of air fed in:	97°C

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35   The parameter K calculated from the equation in claim 1 is 82.9.

At an air flow which is too low, many twin rings and catalysts with flaked coatings were found. The amount of abraded material after the triple drop test (drop test as in example 1) was 64%.

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Comparative example 9:

A two-layer catalyst was prepared as in example 7, with the operating conditions of the fluidized-bed apparatus being set as follows:

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Air flow:	6500 m^3/h
Introduction rate:	2250 g/min
Binder concentration:	7.5% by weight of the total suspension used
Weight of supports:	150 kg of steatite rings (8 mm × 6 mm × 5 mm)
Temperature of air fed in:	125°C

The parameter K calculated from the equation in claim 1 is 212.8.

At a temperature of the air fed in which was too high, many catalysts with flaked coatings were found. The amount of abraded material after the triple drop test (drop test as in example 1) was 65%.

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